

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Sec.

136.1 Applicability.

136.2 Definitions.

136.3 Identification of test procedures.

136.4 Application for alternate test procedures.

136.5 Approval of alternate test procedures.

APPENDIX A TO PART 136—METHODS FOR ORGANIC CHEMICAL ANALYSIS OF MUNICIPAL AND INDUSTRIAL WASTEWATER

APPENDIX B TO PART 136—DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT—REVISION 1.11

APPENDIX C TO PART 136—INDUCTIVELY COUPLED PLASMA—ATOMIC EMISSION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES METHOD 200.7

APPENDIX D TO PART 136—PRECISION AND RECOVERY STATEMENTS FOR METHODS FOR MEASURING METALS

AUTHORITY: Secs. 301, 304(h), 307 and 501(a), Pub. L. 95-217, 91 Stat. 1566, et seq. (33 U.S.C. 1251, et seq.) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

§ 136.1 Applicability.

The procedures prescribed herein shall, except as noted in § 136.5, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(a) An application submitted to the Administrator, or to a State having an approved NPDES program for a permit under section 402 of the Clean Water Act of 1977, as amended (CWA), and/or to reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under parts 122 to 125 of title 40, and,

(b) Reports required to be submitted by discharges under the NPDES established by parts 124 and 125 of this chapter, and,

(c) Certifications issued by States pursuant to section 401 of the CWA, as amended.

[38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 26, 1984]

§ 136.2 Definitions.

As used in this part, the term:

(a) *Act* means the Clean Water Act of 1977, Pub. L. 95-217, 91 Stat. 1566, et seq. (33 U.S.C. 1251 et seq.) (The Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

(b) *Administrator* means the Administrator of the U.S. Environmental Protection Agency.

(c) *Regional Administrator* means one of the EPA Regional Administrators.

(d) *Director* means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) *National Pollutant Discharge Elimination System (NPDES)* means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Act.

(f) *Detection limit* means the minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero as determined by the procedure set forth at appendix B of this part.

[38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 26, 1984]

§ 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in tables IA, IB, IC, ID, and IE. The full text of the referenced test procedures are incorporated by reference into tables IA, IB, IC, ID, and IE. The references and the sources from which they are available are given in paragraph (b) of this section. These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published in the FEDERAL REGISTER. The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in tables IA, IB, IC, ID, and IE, or by any alternate test procedure which has been approved by the Administrator under the provisions of

§ 136.3

paragraph (d) of this section and §§ 136.4 and 136.5 of this part 136. Under certain circumstances (§ 136.3 (b) or (c) or 40 CFR 401.13) other test procedures may be used that may be more advantageous when such other test procedures have been previously approved by

40 CFR Ch. I (7-1-97 Edition)

the Regional Administrator of the Region in which the discharge will occur, and providing the Director of the State in which such discharge will occur does not object to the use of such alternate test procedure.

Environmental Protection Agency

§ 136.3

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS

Parameter and units	Method ¹	EPA	Standard methods, 18th Ed.	ASTM	USGS
Bacteria:					
1. Coliform (fecal), number per 100 mL.	Most Probable Number (MPN), 5 tube 3 dilution, or Membrane filter (MF) ² , single step MPN, 5 tube, 3 dilution, or MF, single step ⁶	p. 132 ³ p. 124 ³ p. 132 ³ p. 124 ³	9221C E ⁴ 9222D ⁴ 9221C E ⁴ 9222D ⁴ B-0050-85 ⁵	
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or MF ² , single step or two step MPN, 5 tube, 3 dilution, or MF ² , with enrichment	p. 114 ³ p. 108 ³ p. 114 ³ p. 111 ³	9221B ⁴ 9222B ⁴ 9221B ⁴ 9222(B+B ₅ C) ⁴ B-0025-85 ⁵	
4. Coliform (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution MF ² , or Plate count	p. 139 ³ p. 136 ³ p. 143 ³	9230B ⁴ 9230C ⁴ B-0056-85 ⁵	
Aquatic Toxicity:					
6. Toxicity, acute, fresh water organisms, LC50, percent effluent.	Daphnia, Cenodaphnia, Fathead Minnow, Rainbow Trout, Brook Trout, or Bannerfish Shiner mortality.	Sec. 97	
7. Toxicity, acute, estuarine and marine organisms, LC50, percent effluent.	Mysid, Sheephead Minnow, or Menidia spp. mortality	Sec. 97	
8. Toxicity, chronic, fresh water organisms, NOEC or IC25, percent effluent.	Fathead minnow larval survival and growth Fathead minnow embryo-larval survival and teratogenicity Ceriodaphnia survival and reproduction Selenastrum growth	1000 ^{0.8} 1001 ^{0.8}	
9. Toxicity, chronic, estuarine and marine organisms, NOEC or IC25, percent effluent.	Sheepshead minnow larval survival and growth Sheepshead minnow embryo-larval survival and teratogenicity Menidia beryllina larval and growth Mysidopsis bahia survival, growth, and fecundity Arbasia punctulata fertilization Champia parvula reproduction	1002 ^{0.8} 1003 ^{0.8} 1004 ^{0.9} 1005 ^{0.9} 1006 ^{0.9} 1007 ^{0.9} 1008 ^{0.9} 1009 ^{0.9}	

Notes to Table IA:

¹The method must be specified when results are reported.
²A 0.45 µm membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

³USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/8-78/017.

⁴APHA. 1992. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, 18th Edition, Amer. Publ. Hlth. Assoc., Washington, DC.

⁵USGS. 1989. U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of Interior, Reston, Virginia.

⁶Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

⁷USEPA. 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Fourth Edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio, August 1993, EPA/600/4-90/027E.

⁸ USEPA, 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Third Edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency USEPA, 1994, Cincinnati, Ohio (July 1994, EPA/600/R-91/002).

⁹ Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Second Edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio (July 1994, EPA/600/R-91/003). These methods do not apply to marine waters of the Pacific Ocean.

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, units and method	EPA ^{1,35}	STD methods 18th ed.	ASTM	USGS ²	Other
1. Acidity as CaCO_3 , mg/L: Electrometric endpoint or phenolphthalein endpoint	305.1 310.1 310.2	2310 B(4a) 2320 B	D1067-92 D1067-92	I-1030-85 I-2030-85 I-3051-85	973.43. ³
2. Alkalinity, as CaCO_3 , mg/L: Electrometric or Colorimetric titration to pH 4.5, manual or automated.	310.1 310.2	2320 B
3. Aluminum—Total, ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration ³⁶ AA furnace Inductively Coupled Plasma/Atomic Emission Spec- trometry (ICP/AES) ³⁶ .	202.1 202.2 5200.7	3111 D 3113 B 3120 B	Note 34.
4. Direct Current Plasma (DCP) ³⁶ Colorimetric (Eriochrome cyanine R)	3500-Al D	D4190-82(88)
5. Ammonia (as N), mg/L: Manual distillation (at pH 9.5), ⁶ followed by Nesslerization Titration Electrode Automated phenate, or Automated electrode	350.2 350.2 350.2 350.3 350.1	4500-NH ₃ B 4500-NH ₃ C 4500-NH ₃ E 4500-NH ₃ F or G 4500-NH ₃ H	D1426-93(A) D1426-93(B)	I-3520-85 I-3520-85 I-4523-85	973.49. ³ 973.49. ³
6. Arsenic—Total, ⁴ mg/L: Digestion ⁴ followed by: AA gaseous hydride AA furnace ICP/AES ³⁶ or Colorimetric (SDDC)	204.1 204.2 5200.7	3111 B 3113 B 3120 B	Note 7.
7. Barium—Total, ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration ³⁶ AA furnace ICP/AES ³⁶ DCP ³⁶	206.5 206.3 5200.7 206.4	3114 B 4.d 3113 B 3120 B 3500-As C	D2972-93(B) D2972-93(C) D2972-93(A)	I-3062-85 I-3060-85
8. Beryllium—Total, ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration AA furnace ICP/AES DCP, or	210.1 210.2 5200.7	3111 D 3113 B 3120 B	D3645-93(88)(A) D3645-93(88)(B) D4190-82(88)	I-3095-85 Note 34.

	Colorimetric (aluminon)	3500-Be D	I-1578-78 ⁸
9.	Biochemical oxygen demand (BOD ₅), mg/L:	405.1 5210 B	I-3112-85
	Dissolved Oxygen Depletion	212.3 4500-B B	D4190-82(88)
10.	Boron ³⁷ —Total, mg/L:	5200.7 3120 B	D1246-82(88)(C)
	Colorimetric (curcumin)	D3557-90(A or B)
	ICP/AES, or DCP	D3557-90(D)
11.	Bromide, mg/L:	320.1	D4190-82(88)
	Titrimetric—Total ⁴ mg/L; Digestion ⁴ followed by:	213.1 3111 B or C	I-1125-85
12.	AA direct aspiration ³⁶	213.2 3113 B	I-3135-85 or I-3136-85
	AA furnace	5200.7 3120 B	I-1472-85
	ICP/AES ³⁶	D4190-82(88)
	DCP ³⁶	D3557-90(C)
13.	Voltammetry, ¹¹ or Colorimetric (Dithizone)	3500-Cd D
	Calcium—Total ⁴ mg/L; Digestion ⁴ followed by:	215.1 3111 B	I-3152-85
	AA direct aspiration	5200.7 3120 B	D611-93(B)
	ICP/AES	D611-93(A)
	DCP, or	215.2 3500-Ca D
14.	Thiometric (EDTA)
	Carbonaceous biochemical oxygen demand (CBOD ₅), mg/L ¹² :	5210 B
	Dissolved Oxygen Depletion with nitrification inhibitor.	410.1 5220 C	D1252-88(A)
15.	Chemical oxygen demand (COD), mg/L; Titrimetric, or.	410.2 5220 D	I-3560-85
	Spectrophotometric, manual or automated	410.3 5220 D	I-3562-85
	Chloride, mg/L:	410.4 5220 D	I-3561-85
16.	Spectrophotometric, manual or automated	4500-Cl ⁻ B	D512-89(B)
	Titrimetric (silver nitrate) or (Mercuric nitrate)	325.3 4500-Cl ⁻ C	D512-89(A)
	Colorimetric, manual or Automated (Fericyanide)	325.1 or 325.2 4500-Cl ⁻ E
17.	Chlorine—Total residual, mg/L; Titrimetric:	330.1 4500-Cl ⁻ D	D1253-86(92)
	Ampereometric direct	330.3 4500-Cl ⁻ B
	Iodometric direct	330.2 4500-Cl ⁻ C
	Back titration ether end-point ¹⁵ or DPD-FAS	330.4 4500-Cl ⁻ F
	Spectrophotometric, DPD	330.5 4500-Cl ⁻ G
	Or Electrode
18.	Chromium VI dissolved, mg/L; 0.45 micron filtration followed by:
	AA chelation-extraction or Colorimetric (Diphenylcarbazide)	218.4 3111 C	D1687-92(A)
19.	Chromium—Total ⁴ mg/L; Digestion ⁴ followed by:	3500-Cr D	D1687-92(B)
	AA direct aspiration ³⁶	218.1 3111 B	I-3236-85
	AA chelation-extraction	218.3 3111 C	D1687-92(C)
	AA furnace	218.2 3113 B

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	EPA 1: ³⁵	STD methods 18th ed.	ASTM	Reference (method number or page)	USGS ²	Other
ICP/AES ³⁶	5200.7	3120 B 3500-Cr D	D4190-82(88)	Note 34.
DCP, ³⁵ or Colorimetric (Diphenylcarbazide)
20. Cobalt—Total, ⁴ mg/L; Digestion, ⁴ followed by: AA direct aspiration	219.1	3111 B or C	D3558-90(A or B)
AA furnace	219.2	3113 B	D3558-90(C)	P. 37. ⁹
ICP/AES	5200.7	3120 B	D4190-82(88)	Note 34.
DCP
21. Color platinum cobalt units or dominant wavelength, hue, luminance purity: Colorimetric (ADM), or (Platinum cobalt), or Spectrophotometric	110.1	2120 E	Note 18.
22. Copper—Total, ⁴ mg/L; Digestion, ⁴ followed by: AA direct aspiration ³⁶	110.2	2120 B
AA furnace	110.3	2120 C	I-1250-85
ICP/AES ³⁶	220.1	3111 B or C	D1688-90(A or B)	974.27. ³ p. 37. ⁹
DCP, ³⁵ or Colorimetric (Neocuproine) or (Bicinchoninate)	220.2	3113 B	D1688-90(C)	I-3270-85 or I-3271-85
23. Cyanide—Total, mg/L: Manual distillation with MgCl ₂ followed by Thiometric, or Spectrophotometric, manual or Automated, ²⁰	220.7	3120 B
.....	3500-Cu D
.....	Or E
.....
24. Cyanide amenable to charring, mg/L: Manual distillation with MgCl ₂ , followed by titrimetric or Spectrophotometric.	31335.2	4500-CN C	D2036-91(A)	P. 22. ⁹
31335.3	4500-CN D
335.1	4500-CN E
335.1	4500-CN F
25. Fluoride—Total mg/L: Manual distillation, ⁶ followed by: Electrode, manual or Automated	340.2	4500-F B	D1179-93(B)	I-4327-85
Colorimetric (SPADNS)	340.1	4500-F C
Or Automated complexone	340.3	4500-F D
26. Gold—Total, ⁴ mg/L; Digestion, ⁴ followed by: AA direct aspiration	231.1	3111 B	Note 34.
AA furnace, or	231.2
DCP
27. Hardness—Total, as CaCO ₃ , mg/L: Automated colorimetric, Titrimetric (EDTA), or Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33).	130.1	2340 B or C	D1126-86(92)	973.52B. ³
28. Hydrogen ion (pH), pH units	130.2

Electrometric measurement, or	150.1	4500-H ⁺ B	D1293-84(90)(A or B)	I-1586-85	973.41. ³ Note 21.
Automated electrode
29. Iridium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration or	235.1	3111 B	D1068-90(A or B)	I-3381-85	974.27. ³
AA furnace	235.2	D1068-90(C)
30. Iron—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	236.1	3111 B or C	D4190-82(88)	Note 34.
AA furnace	236.2	3113 B	D1068-90(D)	Note 22.
ICP/AES ³⁶	5200.7	3120 B
DCP ³⁶ or	3500-Fe D
Colorimetric (Phenanthroline)
31. Kjeldahl Nitrogen—Total, (as N), mg/L;	351.3	4500-NH ₃ B or C	D3590-89(A)	973.48 ₃ .
Digestion and distillation followed by:	351.3	4500-NH ₃ E	D3590-89(A)
Titration	351.3	4500-NH ₃ C	D3590-89(A)
Nesslerization	351.3	4500-NH ₃ F or G
Electrode	351.1
Automated phenate colorimetric	351.2
Semi-automated block digester colorimetric	351.2
Manual or block digester potentiometric	351.4
Block Digester, followed by:
Auto distillation and Titration, or
Nesslerization
Flow injection gas diffusion
32. Lead—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	239.1	3111 B or C	D3559-90(A or B)	I-3399-85	974.27. ³
AA furnace	239.2	3113 B	D3559-90(D)
ICP/AES ³⁶	5200.7	3120 B	D4190-82(88)	Note 34.
DCP ³⁶	D3559-90(C)
Voltammetry ¹¹ or	3500-Pb D
Colorimetric (Dithizone)
33. Magnesium—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	242.1	3111 B	D611-93(B)	I-3447-85	974.27. ³
ICP/AES	5200.7	3120 B	Note 34.
DCP ³⁶ or	3500-Mg D
Gravimetric
34. Manganese—Total, ⁴ mg/L; Digestion ⁴ followed by:					
AA direct aspiration	243.1	3111 B	D858-90(A or B)	I-3454-85	974.27. ³
AA furnace	243.2	3113 B	D858-90(C)
ICP/AES ³⁶	5200.7	3120 B	D4190-82(88)
DCP ³⁶ or	3500-Mn D
Colorimetric (Persulfate), or
(Periodate)
35. Mercury—Total, ⁴ mg/L;					
Cold vapor, manual or	245.1	3112 B	D3223-91	I-3462-85	Note 34, 920-203. ³ Note 23,
Automated	245.2	977.22. ³
36. Molybdenum—Total, ⁴ mg/L; Digestion ⁴ followed by:				
AA direct aspiration	246.1	3111 D	I-3490-85
AA furnace	246.2	3113 B
ICP/AES	5200.7	3120 B

§ 136.3

40 CFR Ch. I (7-1-97 Edition)

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	EPA 1 ³⁵	STD methods 18th ed.	ASTM	Reference (method number or page)	USGS ²	Other
DCP	Note 34.
37. Nickel—Total, ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration ³⁶	249.1 249.2 5200.7	3113 B or C 3113 B	D1886-90(A or B) D1886-90(C)	I-3499-85
AA furnace ICP/AES ³⁶	3120 B	D4190-82(88)
DCP ³⁶ , or Colorimetric (heptoxime)	3500-Ni D
38. Nitrate (as N), mg/L Colorimetric (Brucine sulfate), or Nitrate-nitrite N minus Nitrite N (See Parameters 39 and 40). Nitrate-nitrite (as N), mg/L	362.1	973.50, ³ 419 D, ¹⁷ p. 28, ⁹
Cadmium reduction, Manual or Automated, or Automated hydrazine	363.3 363.2 363.1	4500-NO ₃ ⁻ E 4500-NO ₃ ⁻ F	D3867-90(B) D3867-90(A)	I-4545-85
40. Nitrite (as N), mg/L; Spectrophotometric; Manual or Automated (Diazotization)	364.1	4500-NO ₂ ⁻ B	I-4540-85
41. Oil and grease—Total recoverable, mg/L; Gravimetric (extraction)	413.1	5520 B ₃₈	973.47, ³ p. 14. ²⁴
42. Organic carbon—Total (TOC), mg/L; Combustion or oxidation	415.1	5310 B, C, or D	D2579-93 (A or B)
43. Organic nitrogen (as N), mg/L; Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4)
44. Orthophosphate (as P), mg/L; Ascorbic acid method; Automated, or Manual two reagent	365.1 365.2 365.3	4500-P F 4500-P E	D515-88(A)	I-4601-85	973.56, ³ 973.55, ³
45. Osmium—Total ⁴ , mg/L; Digestion ⁴ followed by: AA direct aspiration, or	252.1 252.2	3111 D	973.45B, ³
AA furnace
46. Oxygen dissolved, mg/L; Winkler (Azide modification), or	360.2 360.1	4500-O C 4500-O G	D888-92(A) D888-92(B)	I-1575-78 ^s I-1576-78 ^s	p. S27, ¹⁰ p. S28, ¹⁰ Note 34.
Electrode
47. Palladium—Total ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration, or	253.1 253.2	3111 B
AA furnace
DCP
48. Phenols, mg/L; Manual distillation ²⁶	420.1	Note 27.
Followed by: Colorimetric (AAP) manual, or Automated ¹⁹	420.1 420.2	Note 27.
49. Phosphorus (elemental), mg/L;

Gas-liquid chromatography	Note 28.
Phosphorus—Total, mg/L	973.55. ³
Persulfate digestion followed by:	973.55. ³
Manual or	973.56. ³
Automatic ascorbic acid reduction	365.2	973.56. ³
Semi-automated block digester	365.3	973.56. ³
Platinum—Total, 4 mg/L; Digestion ⁴ followed by:	365.4	973.56. ³
AA direct aspiration	255.1
AA furnace	255.2
DCP	3111 B
Potassium—Total, 4 mg/L; Digestion ⁴ followed by:	365.5
AA direct aspiration	258.1
ICP/AES	200.7
Flame photometric, or	3111 B
Colometric	3120 B
Residue—Total, mg/L	3500-K D
Gravimetric, 103–105°	160.3
Residue—filterable, mg/L	2540 B
Gravimetric, 180°	160.1
Residue—nonfilterable (TSS), mg/L	2540 C
Gravimetric, 103–105° post washing of residue	160.2
Residue settleable, mg/L	2540 D
Volumetric, (Imhoff cone), or gravimetric	160.5
Residue—Volatile, mg/L	2540 F
Gravimetric, 550°	160.4
Rhodium—Total, 4 mg/L; Digestion ⁴ followed by:	265.1
AA direct aspiration, or	265.2
AA furnace	3111 B
Ruthenium—Total, 4 mg/L; Digestion ⁴ followed by:	267.1
AA direct aspiration, or	267.2
Selenium—Total, 4 mg/L; Digestion ⁴ followed by:	270.2
AA furnace	200.7
ICP/AES ³⁶ or	3113 B
AA gaseous hydride	3120 B
Silica ³⁷ —Dissolved, mg/L; 0.45 micron filtration followed by:	3114 B
Colometric, Manual or	D3859-93(A)
Automated (Molybdate), or	D3859-93(B)
ICP	4500-SI D
Silver—Total, 4 mg/L; Digestion ^{4,29} followed by:	370.1
AA direct aspiration	200.7
AA furnace	3120 B
ICP/AES	272.1
DCP	3111 B or C
Sodium—Total, 4 mg/L; Digestion ⁴ followed by:	272.2
AA direct aspiration	200.7
AA furnace	3113 B
ICP/AES	3120 B
Sodium—Total, 4 mg/L; Digestion ⁴ followed by:	273.1
AA direct aspiration	200.7
DCP, or	3111 B
ICP/AES	3120 B

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	EPA 1 ³⁵	STD methods 18th ed.	ASTM	Reference (method number or page)	USGS ²	Other
Flame photometric	3500 Na D
Specific conductance, micromhos/cm at 25 °C:	120.1 2510 B	D1125-91(A)	I-1780-85	973.40. ³
Wheatstone bridge
Sulfate (as SO ₄) mg/L:	375.1 4500-SO ₄ -2 C or D	925.54. ³
Automated colorimetric (barium chloranilate)	375.3 4500-SO ₄ -2 C or D	426C. ₃₀
Gravimetric	375.4 4500-SO ₄ -2 C or D
Turbidimetric, or
Sulfide (as S), mg/L:
Titrimetric (iodine) or	376.1 4500-S ⁻² E
Colorimetric (methylene blue)	376.2 4500-S ⁻² D
Sulfite (as SO ₃), mg/L:	377.1 4500-SO ₃ ⁻² B
Titrimetric (iodine-iodate)	425.1 5540 C	D2330-88
Surfactants, mg/L:
Colorimetric (methylene blue)
Temperature, °C:	170.1 2550 B	Note 32.
Thermometric
70. Thallium—Total, ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration	279.1 3111 B
AA furnace	279.2 3120 B
ICP/AES, or	5200.7 3120 B
71. Tin—Total, ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration	282.1 3111 B
AA furnace, or	282.2 3113 B
ICP/AES	5200.7 3113 B
72. Titanium—Total, ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration	283.1 3111 D
AA furnace	283.2 3111 D
DCP
Turbidity, NTU:	180.1 2130 B
Nephelometric	D1889-88(A)	I-3860-85
74. Vanadium—Total, ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration	286.1 3111 D
AA furnace	286.2 3120 B	D3373-93
ICP/AES	5200.7 3120 B
DCP, or	D4190-82(88)
Colorimetric (Gallic acid)
Zinc—Total, ⁴ mg/L; Digestion ⁴ followed by: AA direct aspiration ³⁶	289.1 3111 B or C	D1691-90 (A or B)	I-3900-85
AA furnace	289.2 3120 B
ICP/AES ³⁶	5200.7 3120 B
DCP, ³⁶ or	D4190-82(88)
Colorimetric (Dithizone) or	3500-Zn E	Note 34.

Environmental Protection Agency

§ 136.3

<p>Table IB Notes:</p> <p>1. "Methods for Chemical Analysis of Water and Wastes", Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-CI), EPA-600/4-79-020, Revised March 1983 and 1979 where applicable.</p> <p>2. Fishman, M.J., et al. "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, Techniques of Water—Resource Investigations of the U.S. Geological Survey, Denver, CO, Revised 1985, unless otherwise stated.</p> <p>3. Official Methods of Analysis of the Association of Official Analytical Chemists," methods manual, 15th ed. (1990).</p> <p>4. For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979 and 1983". One (section 4.1.3.) is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (section 4.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all samples types. If a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds are broken so that the metal is in a reactive state. In those situations the analyst may also benefit by this vigorous digestion, however, a vigorous digestion with concentrated nitric acid will convert antimony and tin to insoluble oxides and render them unavailable for analysis. Use of ICPAES as well as the method write-up should be consulted for specific instructions and/or cautions.</p> <p>NOTE TO TABLE IB Note 4: If the digestion procedure for direct aspiration AA included in one of the other approved references is different than the above, the EPA procedure must be used.</p> <p>Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion of the filtrate for dissolved metals (or digestion of the original sample solution for total metals) may be omitted for AA (direct aspiration or graphite furnace ICP analyses), provided the sample solution to be analyzed meets the following criteria.</p> <ul style="list-style-type: none"> a. has a low COD (<20) b. is visibly transparent with a turbidity of 1 NTU or less c. is colorless with no perceptible odor, and d. is of one liquid phase and free of particulate or suspended matter following acidification. <p>5 The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 36.</p> <p>6 Manual distillation is not required if comparability data are on company file to show that this preliminary distillation step is not necessary; however, a manual distillation will be required to resolve any controversies.</p> <p>7 Ammonia, Elmsford, NY 10523.</p> <p>8 The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979).</p> <p>9 American National Standard on Photographic Processing of Effluents, Apr. 2, 1975, Available in ANSI/1430 Broadway, New York, NY 10018.</p> <p>10 "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).</p> <p>11 The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.</p> <p>12 Carboxonaceous biochemical oxygen demand (BOD₅) must not be confused with the traditional BOD₅ test, which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CEOD₅ parameter. A discharger whose permit requires reporting the traditional BOD₅ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CEOD₅ is required can the permittee report data using the nitrification inhibitor.</p> <p>13 OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.</p> <p>14 Chemical Oxygen Demand Method 8000, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.</p> <p>15 The back filtration method will be used to resolve any controversy.</p> <p>16 Orion Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138. The calibration graph on the Orion residual chlorine method must be derived using a reagent blank and three standard solutions, containing 0.2, 1.0, and 5.0 ml 0.0281 N potassium iodate/100 ml solution, respectively.</p> <p>17 The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976.</p> <p>18 National Council of the Paper Industry for Air and Stream Improvement, Inc., Technical Bulletin 253, December 1971.</p> <p>19 Copper, Biocinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.</p> <p>20 After the manometric distillation is completed, the autoanalyzers manifolds in EPA Method 335.3 (cyanide) or 402.2 (phenols) are simplified by connecting the re-sample line directly to the sample manifold.</p> <p>21 Hydrogen on (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II.</p> <p>22 Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Water Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, CO 80537.</p> <p>23 Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Water Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.</p> <p>24 Wierschaw, R.L., et al., "Methods for Analysis of Organic Substances in Water," Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972) Revised 1987, p. 14.</p> <p>25 Nitrogen, Nitrite Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.</p>	<p>(Zircon) 350-Zn F Note 33.</p>
--	---

§ 136.3

40 CFR Ch. I (7-1-97 Edition)

²⁷The approved method is cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0±0.2. The approved methods are given on pp 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure.

²⁸R. F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Journal of Chromatography, vol. 47, No. 3, pp. 421-426, 1970.

²⁹Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/l and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/l, 20 ml of sample should be diluted to 100 ml by adding 40 ml each of 2 M Na₂S₂O₃ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/l the approved method is satisfactory.

³⁰The approved method is cited in Standard Methods for the Examination of Water and Wastewater to be adjusted to 0.25 N before colorimetric determination of total cyanide.

³¹EPA Methods 335.2 and 335.3 require the NaOH adsorber solution final concentration to be adjusted to 0.25 N before colorimetric determination of total cyanide.

³²Stevens, H.H., Fricke, J.F., and Snoot, G.F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation", Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975.

³³Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333; Hach Chemical Company, Loveland, CO 80537.

³⁴"Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AE0029," 1986—Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923.

³⁵Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of this part titled, "Precision and Recovery Statements for Methods for Measuring Metals".

³⁶Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, P O Box 200, Matthews, NC 28106-0200, April 16, 1992. Available from the CEM Corporation.

³⁷When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.

³⁸Only the tetrachloroethane extraction solvent is approved.

³⁹Nitrogen, Total Kjeldahl, Method PA-I-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, Perstop Analytical Corporation.

⁴⁰Nitrogen, Total Kjeldahl, Method PA-I-DK02 (Block Digestion, Steam Distillation, Colorimetric Detection), revised 12/22/94, Perstop Analytical Corporation.

⁴¹Nitrogen, Total Kjeldahl, Method PA-I-DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, Perstop Analytical Corporation.

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter ¹	GC	GC/MS	HPLC	Standard method 18th Ed.	ASTM	Other
1. Acenaphthene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
2. Acenaphthylene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
3. Acrolein	603	4604, 1624		
4. Acrylonitrile	603	4624, 1624	610	6410 B, 6440 B	D4657-92	
5. Anthracene	610	625, 1625	610	6410 B, 6440 B	D4657-92	Note 3, p 1.
6. Benzene	602	624, 1624	6210 B, 6220 B		
7. Benzidine	6225, 1625	605	D4657-92	
8. Benzo(a)anthracene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
9. Benzo(a)pyrene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
10. Benzo(d)fluoranthene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
11. Benz(g, h, i)perylene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
12. Benz(k)fluoranthene	610	625, 1625	610	6410 B, 6440 B	D4657-92	
13. Benzyl chloride	Note 3, p 130; Note 6, p. S102.
14. Benzyl butyl phthalate	606	625, 1625	6410 B		
15. Bis(2-chloroethoxy) methane	611	625, 1625	6410 B		
16. Bis(2-chloroethyl) ether	611	625, 1625	6410 B		
17. Bis(2-ethylhexyl) phthalate	606	625, 1625	6410 B, 6230 B		
18. Bromodichloromethane	601	624, 1624	6210 B, 6230 B		
19. Bromoform	601	624, 1624	6210 B, 6230 B		
20. Bromomethane	601	624, 1624	6210 B, 6230 B		
21. 4-Bromophenylphenyl ether	611	625, 1625	6410 B		

Environmental Protection Agency

§ 136.3

22. Carbon tetrachloride	601	624, 1624	6230 B, 6410 B	Note 3, p.130.
23. 4-Chloro-3-methylphenol	604	625, 1625	6410 B, 6420 B	
24. Chlorobenzene	602	624, 1624	6210 B, 6220 B	Note 3, p.130.
25. Chloroethane	601	624, 1624	6210 B, 6230 B	
26. 2-Chloroethylvinyl ether	601	624, 1624	6210 B, 6230 B	
27. Chloroform	601	624, 1624	6210 B, 6230 B	Note, p.130.
28. Chloromethane	601	624, 1624	6210 B, 6230 B	
29. 2-Chlorophthalalene	612	625, 1625	6410 B	
30. 2-Chlorophenol	604	625, 1625	6410 B, 6420 B	
31. 4-Chlorophenylphenyl ether	611	625, 1625	6410 B	
32. Chrysene	610	625, 1625	6410 B, 6440 B	
33. Dibenz(a,h)anthracene	610	625, 1625	6410 B, 6440 B	
34. Dibromo-chloromethane	601	624, 1624	6210 B, 6230 B	
35. 1, 2-Dichlorobenzene	601, 602, 612	624, 625, 1625	6410 B, 6230 B, 6220 B	
36. 1, 3-Dichlorobenzene	601, 602, 612	624, 625, 1625	6410 B, 6220 B, 6230 B	
37. 1,4-Dichlorobenzene	601	625, 1625	6410 B	
38. 3, 3-Dichlorobenzidine	601	605	6410 B	
39. Dichlorodifluoromethane	601	6230 B	
40. 1, 1-Dichloroethane	601	624, 1624	6230 B, 6210 B	
41. 1, 2-Dichloroethane	601	624, 1624	6230 B, 6210 B	
42. 1, 1-Dichloroethane	601	624, 1624	6230 B, 6210 B	
43. trans-1, 2-Dichloroethylene	601	624, 1624	6230 B, 6210 B	
44. 2, 4-Dichlorophenol	604	625, 1625	6420 B, 6410 B	
45. 1, 2-Dichloropropane	601	624, 1624	6230 B, 6210 B	
46. cis-1, 3-Dichloropropene	601	624, 1624	6230 B, 6210 B	
47. trans-1, 3-Dichloropropene	601	624, 1624	6230 B, 6210 B	
48. Diethyl phthalate	606	625, 1625	6410 B	
49. 2, 4-Dimethylphenol	604	625, 1625	6420 B, 6410 B	
50. Dimethyl phthalate	606	625, 1625	6410 B	
51. Di-n-butyl phthalate	606	625, 1625	6410 B	
52. Di-n-octyl phthalate	606	625, 1625	6410 B	
53. 2, 3-Dinitrophenol	604	625, 1625	6420 B, 6410 B	
54. 2, 4-Dinitrotoluene	609	625, 1625	6410 B	
55. 2, 6-Dinitrotoluene	609	625, 1625	6410 B	
56. Epichlorohydrin	Note 3, p.130 Note 6, p.S102.
57. Ethylbenzene	602	624, 1624	6220 B, 6210 B	
58. Fluoranthene	610	625, 1625	6410 B, 6440 B	D4657-92 D4657-92
59. Fluorene	610	625, 1625	6410 B, 6440 B	
60. Hexachlorobenzene	612	625, 1625	6410 B	
61. Hexachlorobutadiene	612	625, 1625	6410 B	
62. Hexachlorocyclopentadiene	612	625, 1625	6410 B	
63. Hexachloroethane	616	625, 1625	6410 B	
64. Idenol(1,2-3-d) pyrene	610	625, 1625	6410 B, 6440 B	D4657-92
65. Isophorone	609	625, 1625	6410 B	
66. Methylene chloride	601	624, 1624	6230 B	
67. 2-Methyl-4, 6-dinitrophenol	604	625, 1625	6420 B, 6410 B	
68. Naphthalene	610	625, 1625	6410 B, 6440 B	

§ 136.3

40 CFR Ch. I (7-1-97 Edition)

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter ¹	EPA method number ²					ASTM	Other
	GC	GC/MS	HPLC	Standard method 18th Ed.			
69. Nitrobenzene	609	625, 1625	6410 B		
70. 2-Nitrophenol	604	625, 1625	6410 B, 6420 B		
71. 4-Nitrophenol	604	625, 1625	6410 B, 6420 B		
72. N-Nitrosodimethylamine	607	625, 1625	6410 B		
73. N-Nitrosodimethylamine	607	5625, 1625	6410 B		
74. N-Nitrosodipropylamine	607	625, 1625	6410 B		
75. 2,2-Oxybis(1-chloropropane)	611	625, 1625	6410 B		
76. PCB-1016	608	625	6410 B		
77. PCB-1221	608	625	6410 B		
78. PCB-1232	608	625	6410 B		
79. PCB-1242	608	625	6410 B		
80. PCB-1248	608	625	6410 B		
81. PCB-1254	608	625	6410 B		
82. PCB-1260	608	625	6410 B, 6630 B		
83. Pentachlorophenol	604	625, 1625	6410 B, 6630 B		
84. Phenanthrene	610	625, 1625	6410 B, 6630 B		
85. Phenol	604	625, 1625	6410 B, 6640 B		
86. Pyrene	610	625, 1625	6420 B, 6410 B		
87. 2,3,7,8-Tetrachlorobenzo-p-dioxin	6410 B, 6440 B		
88. 1,1,2,2-Tetrachloroethane	601	624, 1624	6230 B, 6210 B		
89. Tetrachloroethylene	601	624, 1624	6230 B, 6210 B		
90. Toluene	602	624, 1624	6210 B, 6220 B		
91. 1,2,4-Trichlorobenzene	612	625, 1625	6410 B		
92. 1,1,1-Trichloroethane	601	624, 1624	6210 B, 6230 B		
93. 1,1,2-Trichloroethane	601	624, 1624	6210 B, 6230 B		
94. Trichloroethylene	601	624, 1624	6210 B, 6230 B		
95. Trichlorofluoromethane	604	625, 1625	6410 B, 6240 B		
96. 2,4,6-Trichlorophenol	601	624, 1624	6210 B, 6230 B		
97. Vinyl chloride							

Table IC notes:

¹ All parameters are expressed in micrograms per liter ($\mu\text{g/L}$).
² The full text of Methods 601-613, 624, 625, 1624, and 1625, are given at appendix A, "Test Procedures for Analysis of Organic Pollutants," of this part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at appendix B, "Definition and Procedure for the Determination of the Method Detection Limit" of this part 136.

³ Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater." U.S. Environmental Protection Agency, September, 1978.
⁴ Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624.

Environmental Protection Agency

§ 136.3

⁵Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.

^{5.1}Method 625, Screening only.

⁶"Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

⁷Each Analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 624, 625, 1624, and 1625 (See Appendix A of this Part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 624 and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods.

⁸Note: These warning limits are promulgated as an "interim final action with a request for comments."

^{8.0}Organochlorine Pesticides and PCBs in Wastewater Using Empore TM Disk™, 3M Corporation Revised 10/28/94.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES¹

Parameter	Method	EPA ² 7	Standard methods 18th Ed.	ASTM	Other
1. Aldrin	GC GC/MSS GC TLC	608 625	6630 B & C 6410 B	D3036-90	Note 3, p. 7; note 4, p. 30; note 8.
2. Ametryn	GC	Note 3, p. 83; Note 6, p. S68.
3. Aminocab	GC	Note 3, p. 94; Note 6, p. S16.
4. Atralon	GC	Note 3, p. 83; Note 6, p. S68.
5. Atrazine	GC	Note 3, p. 83; Note 6, p. S68.
6. Azinphos methyl	GC TLC 608 6630 B & C	Note 3, p. 25; Note 6, p. S51.
7. Barban	GC	5625	6410 B	D3036-90	Note 3, p. 104; Note 6, p. S64.
8. α -BHC	GC	608	6630 C	D3036-90	Note 3, p. 7; note 8.
9. β -BHC	GC	5625	6410 B	D3036-90	Note 8.
10. δ -BHC	GC GC/MSS GC	608 5625 608	6630 C .. 6410 B .. 6630 B & C ..	D3036-90 .. D3036-90 .. D3036-90 ..	Note 8.
11. δ -BHC (Lindane)	GC GC/MSS	625	6410 B	Note 3, p. 7; note 4, p. 30; note 8.
12. Captan	GC	D3036-90 ..	Note 3, p. 7.
13. Carbaryl	TLC	Note 3, p. 94; Note 6, p. S60.
14. Carbofenthion	GC	608	6630 B & C ..	D3036-90 ..	Note 4, p. 30; Note 6, p. S73.
15. Chlordane	GC/MSS	625	6410 B	Note 3, p. 7; note 8.
16. Chloropropham	TLC	Note 3, p. 104; Note 6, p. S64.
17. 2,4-D	GC	Note 3, p. 115; Note 4, p. 35.
18. 4,4'-DDD	GC GC/MSS	608 625	6630 B & C .. 6410 B ..	D3036-90 .. D3036-90 ..	Note 3, p. 7; note 4, p. 30; note 8.
19. 4,4'-DDE	GC GC/MSS	608 625	6630 B & C .. 6410 B ..	D3036-90 .. D3036-90 ..	Note 3, p. 7; note 4, p. 30; note 8.
20. 4,4'-DDT	GC GC/MSS	608 625	6630 B & C .. 6410 B ..	D3036-90 .. D3036-90 ..	Note 3, p. 7; note 4, p. 30; note 8.
21. Demeton-O	GC	Note 3, p. 25; Note 6, p. S51.
22. Demeton-S	GC	Note 3, p. 25; Note 6, p. S51.

§ 136.3

40 CFR Ch. I (7-1-97 Edition)

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES 1—Continued

Parameter	Method	EPA ²⁷	Standard methods 18th Ed.	ASTM	Other
23. Diazinon	GC	Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
24. Dicamba	GC	Note 3, p. 115.
25. Dichofenthion	GC	Note 4, p. 30; Note 6, p. S73.
26. Dichloran	GC	D3086-90	Note 3, p. 7.
27. Dicofol	GC	608	6630 B & C	Note 3, p. 7; note 4, p. 30; note 8.
28. Dieldrin	GC/MSS	625	6410 B
29. Dioxathion	GC	Note 4, p. 30; Note 6, p. S73.
30. Disulfoton	GC	Note 3, p. 25; Note 6, p. S51.
31. Diuron	TLC	608	6630 B & C	D3086-90	Note 3, p. 104; Note 6, p. S64.
32. Endosulfan I	GC/MSS	5625	6410 B	Note 3, p. 7; note 8.
33. Endosulfan II	GC/MSS	608	6630 B & C	D3086-90
34. Endosulfan Sulfate	GC	608	6630 C
35. Endrin	GC/MSS	625	6410 B
36. Endrin aldehyde	GC	608	6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 8.
37. Ethion	GC	625	6410 B	Note 8.
38. Fenuron	TLC
39. Fenuron-TCA	TLC	608	6630 B & C	D3086-90
40. Heptachlor	GC	625	6410 B
41. Heptachlor epoxide	GC/MSS	608	6630 B & C	D3086-90	Note 3, p. 7; note 4, p. 30; note 6, p. S73; note 8.
42. Isodrin	GC	625	6410 B
43. Linuron	GC	Note 4, p. 30; Note 6, p. S73.
44. Malathion	GC	Note 3, p. 104; Note 6, p. S64.
45. Methiocarb	TLC	608	6630 C	Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
46. Methoxychlor	GC	D3086-90	Note 3, p. 94; Note 6, p. S60.
47. Mexacarbate	TLC	Note 3, p. 7; note 4, p. 30; note 8.
48. Mirex	GC
49. Monuron	TLC	Note 3, p. 104; Note 6, p. S64.
50. Monuron	TLC	Note 3, p. 104; Note 6, p. S64.
51. Nuburon	GC	Note 3, p. 25; Note 4, p. 30.
52. Parathion methyl	GC	6630 C	Note 3, p. 25.
53. Parathion ethyl	GC	6630 C	Note 3, p. 7.
54. PCNB	GC	6630 B & C

		D3086-90
55. Perthane	GC
56. Prometon	GC	Note 3, p. 83; Note 6, p. S68.
57. Prometryn	GC	Note 3, p. 83; Note 6, p. S68.
58. Propazine	GC	Note 3, p. 83; Note 6, p. S68.
59. Propham	TLC	Note 3, p. 104; Note 6, p. S64.
60. Propoxur	TLC	Note 3, p. 94; Note 6, p. S60.
61. Secbuturon	TLC	Note 3, p. 83; Note 6, p. S68.
62. Siduron	TLC	Note 3, p. 104; Note 6, p. S64.
63. Simazine	GC	Note 3, p. 83; Note 6, p. S68.
64. Stroboane	GC	Note 3, p. 7.
65. Swep	TLC	Note 3, p. 104; Note 6, p. S64.
66. 2,4,5-T ^a	GC	Note 3, p. 115; Note 4, p. 35.
67. 2,4,5-TP (Silvex)	GC	Note 3, p. 115
68. Terbutylazine	GC	Note 3, p. 83; Note 6, p. S68.
69. Toxaphene	GC/MS	D3086-90
70. Trifluralin	GC	Note 3, p. 7; note 4, p. 30; note 8. Note 3, p. 7.

Table ID notes:

¹Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 1C, where entries are listed by chemical name.

²The full text of Methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit" of this Part 136.

³"Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.

⁴"Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3" (1987).

⁵The method may be extended to include α -BHC, γ -BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

⁶"Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

⁷Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625. (See Appendix A of this Part 136) in accordance with procedures given in section 8.2 of each of these methods. Additionally, each laboratory, on an-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other Methods cited.

⁸NOTE: These warning limits are promulgated as an "Inherent final action with a request for comments."

⁹"Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk", 3M Corporation, Revised 10/28/94.

TABLE IE.—LIST OF APPROVED RADIOLOGIC TEST PROCEDURES

Parameter and units	Method	Reference (method number or page)		
		EPA ¹	Standard meth-ods 18th Ed.	ASTM
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900	7110 B	D1943-90
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	7110 B	D1943-90
3. Beta-Total, pCi per liter	Proportional counter	9000	7110 B	D1890-90
4. Beta-Counting error, pCi	Proportional counter	7110 B	D1890-90
5. (a) Radium Total pCi per liter	Proportional counter	903.0	7500Ra B	D2460-90
(b) Ra, pCi per liter	Scintillation counter	903.1	7500Ra C	D3454-91

Table IE notes:

¹ Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980), U.S. Environmental Protection Agency, August 1980.² Fishman, M.J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).³ The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total".

Environmental Protection Agency

§ 136.3

(b) The full texts of the methods from the following references which are cited in Tables IA, IB, IC, ID, and IE are incorporated by reference into this regulation and may be obtained from the sources identified. All costs cited are subject to change and must be verified from the indicated sources. The full texts of all the test procedures cited are available for inspection at the Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, 26 West Martin Luther King Dr., Cincinnati, OH 45268 and the Office of the Federal Register, room 8301, 1110 L Street, NW., Washington, DC 20408.

REFERENCES, SOURCES, COSTS, AND TABLE CITATIONS:

(1) The full text of Methods 601-613, 624, 625, 1624, and 1625 are printed in appendix A of this part 136. The full text for determining the method detection limit when using the test procedures is given in appendix B of this part 136. The full text of Method 200.7 is printed in appendix C of this part 136. Cited in: Table IB, Note 5; Table IC, Note 2; and Table ID, Note 2.

(2) USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/8-78/017. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB-290329/AS. Cost: \$36.95. Table IA, Note 3.

(3) "Methods for Chemical Analysis of Water and Wastes," U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1979, or "Methods for Chemical Analysis of Water and Wastes," U.S. Environmental Protection Agency, EPA-600/4-79-020, Revised March 1983. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IB, Note 1.

(4) "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, 1978. Available from: ORD Publications, CERI,

U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IC, Note 3; Table D, Note 3.

(5) "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," U.S. Environmental Protection Agency, EPA-600/4-80-032, 1980. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IE, Note 1.

(6) American Public Health Association. 1992. Standard Methods for the Examination of Water and Wastewater. 18th Edition. Amer. Publ. Hlth. Assoc., 1015 15th Street NW, Washington, DC 20005. Cost: \$160.00. Table IA, Note 4.

(7) Ibid, 15th Edition, 1980. Table IB, Note 30; Table ID.

(8) Ibid, 14th Edition, 1975. Table IB, Notes 17 and 27.

(9) "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the 15th Edition of Standard Methods for the Examination of Water and Wastewater, 1981. Available from: American Public Health Association, 1015 Fifteenth Street NW., Washington, DC 20036. Cost available from publisher. Table IB, Note 10; Table IC, Note 6; Table ID, Note 6.

(10) Annual Book of ASTM Standards, Water and Environmental Technology, Section 11, Volumes 11.01 and 11.02, 1994 in 40 CFR 136.3, Tables IB, IC, ID and IE.

(11) USGS. 1989. U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of the Interior, Reston, Virginia. Available from: USGS Books and Open-File Reports Section, Federal Center, Box 25425, Denver, Colorado 80225. Cost: \$18.00. Table IA, Note 5.

(12) "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," by M.J. Fishman and Linda C. Friedman, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5 Chapter A1 (1989). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$108.75 (subject to change). Table IB, Note 2.

§ 136.3

40 CFR Ch. I (7-1-97 Edition)

(13) "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," N.W. Skougstad and others, editors. Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1 (1979). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$10.00 (subject to change), Table IB, Note 8.

(14) "Methods for the Determination of Organic Substances in Water and Fluvial Sediments," Wershaw, R.L., et al, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$0.90 (subject to change). Table IB, Note 24; Table ID, Note 4.

(15) "Water Temperature—Influential Factors, Field Measurement and Data Presentation," by H.H. Stevens, Jr., J. Ficke, and G.F. Smoot, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$1.60 (subject to change). Table IB, Note 32.

(16) "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," by M.J. Fishman and Eugene Brown; U.S. Geological Survey Open File Report 76-77 (1976). Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Cost: \$13.50 (subject to change). Table IE, Note 2.

(17) "Official Methods of Analysis of the Association of Official Analytical Chemists", Methods manual, 15th Edition (1990). Price: \$240.00. Available from: The Association of Official Analytical Chemists, 2200 Wilson Boulevard, Suite 400, Arlington, VA 22201. Table IB, Note 3.

(18) "American National Standard on Photographic Processing Effluents," April 2, 1975. Available from: American National Standards Institute, 1430 Broadway, New York, New York 10018. Table IB, Note 9.

(19) "An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color," NCASI Technical Bulletin No. 253, December 1971. Available from: National

Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, NY 10016. Cost available from publisher. Table IB, Note 18.

(20) Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976. Technicon Auto Analyzer II. Method and price available from Technicon Industrial Systems, Tarrytown, New York 10591. Table IB, Note 7.

(21) Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Method price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 14.

(22) OIC Chemical Oxygen Demand Method, 1978. Method and price available from Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840. Table IB, Note 13.

(23) ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Method and price available from ORION Research Incorporation, 840 Memorial Drive, Cambridge, Massachusetts 02138. Table IB, Note 16.

(24) Bicinchoninate Method for Copper. Method 8506, Hach Handbook of Water Analysis, 1979, Method and price available from Hach Chemical Company, P.O. Box 300, Loveland, Colorado 80537. Table IB, Note 19.

(25) Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA. October 1976. Bran & Luebbe (Technicon) Auto Analyzer II. Method and price available from Bran & Luebbe Analyzing Technologies, Inc. Elmsford, N.Y. 10523. Table IB, Note 21.

(26) 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water, Hach Method 8008, 1980. Method and price available from Hach Chemical Company, P.O. Box 389 Loveland, Colorado 80537. Table IB, Note 22.

(27) Periodate Oxidation Method for Manganese, Method 8034, Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 23.

(28) Nitrogen, Nitrite—Low Range, Diazotization Method for Water and Wastewater, Hach Method 8507, 1979.

Environmental Protection Agency

§ 136.3

Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 25.

(29) Zincon Method for Zinc, Method 8009. Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 33.

(30) "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," by R.F. Addison and R.G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421-426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164. Cost available from publisher. Table IB, Note 28.

(31) "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes", Method AES 0029, 1986-Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923. Table B, Note 34.

(32) "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals, CEM Corporation, P.O. Box 200, Matthews, North Carolina 28106-0200, April 16, 1992. Available from the CEM Corporation. Table IB, Note 36.

(33) "Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk" Test Method 3M 0222, Revised 10/28/94. 3M Corporation, 3M Center Building 220-9E-10, St. Paul, MN 55144-1000. Method available from 3M Corporation. Table IC, Note 8 and Table ID, Note 8.

(34) USEPA. 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Fourth Edition, December 1993. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio (EPA/600/4-90/027F). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB-91-167650. Cost: \$31.00. Table IA, Note 17. See changes in the manual, listed in Part V of this rule.

(35) "Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection)", revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 39.

(36) "Nitrogen, Total Kjeldahl, Method PAI-DK02 (Block Digestion, Steam Distillation, Colorimetric Detection)", revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 40.

(37) "Nitrogen, Total Kjeldahl, Method PAI-DK03 (Block Digestion, Automated FIA Gas Diffusion)", revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 41.

(38) USEPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Third Edition. July 1994. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. (EPA/600/4-91/002). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB-92-139492. Cost: \$31.00. Table IA, Note 8.

(39) USEPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Second Edition, July 1994. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/4-91/003. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB-92-139484. Cost: \$45.00. Table IA, Note 9.

(c) Under certain circumstances the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator, or the Director

§ 136.3

40 CFR Ch. I (7-1-97 Edition)

upon the recommendation of the Director of the Environmental Monitoring Systems Laboratory—Cincinnati.

(d) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring Systems Laboratory—Cincinnati, additional alternate test procedures for nationwide use.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters cited in Tables IA, IB, IC, ID, and IE are prescribed in Table II. Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters

to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded, by the Regional Administrator, to the Director of the Environmental Monitoring Systems Laboratory—Cincinnati, Ohio for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Director of the Environmental Monitoring Systems Laboratory, the Regional Administrator may grant a variance applicable to the specific charge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
Table IA—Bacteria Tests:			
1–4 Coliform, fecal and total	P, G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₅ ⁵ ...	6 hours.
5 Fecal streptococci	P, G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₅ ⁵ ...	6 hours.
Table IA—Aquatic Toxicity Tests:			
6–10 Toxicity, acute and chronic	P, G	Cool, 4°C ¹⁶	6 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, Gdo	Do.
4. Ammonia	P, G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
10. Boron	P, PTFE, or Quartz.	HNO ₃ , TO pH<2	6 months.
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, Gdo	Analyze immediately.
21. Color	P, G	Cool, 4°C	48 hours.
23–24. Cyanide, total and amenable to chlorination.	P, G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid ⁵ .	14 days. ⁶
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO ₃ to pH<2, H ₂ SO ₄ to pH<2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.
Metals: ⁷			
18. Chromium VI	P, G	Cool, 4°C	24 hours.
35. Mercury	P, G	HNO ₃ to pH<2	6 months.
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except boron, chromium VI and mercury.	P, Gdo	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite	P, G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool to 4°C, HCl or H ₂ SO ₄ to pH<2	28 days.
42. Organic Carbon	P, G	Cool to 4 °C HC1 or H ₂ SO4 or H ₃ PO4, to pH<2	28 days.
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C ..	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top.	None required	Analyze immediately.
47. Winklerdo	Fix on site and store in dark ...	8 hours.
48. Phenols	G only	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.

Environmental Protection Agency

§ 136.3

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter No./name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, G	do	7 days.
55. Residue, Nonfilterable (TSS)	P, G	do	7 days.
56. Residue, Settleable	P, G	do	48 hours.
57. Residue, volatile	P, G	do	7 days.
61. Silica	P, PTFE, or Quartz.	Cool, 4 °C	28 days.
64. Specific conductance	P, G	do	Do.
65. Sulfate	P, G	do	Do.
66. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9.	7 days.
67. Sulfite	P, G	None required	Analyze immediately.
68. Surfactants	P, G	Cool, 4°C	48 hours.
69. Temperature	P, G	None required	Analyze.
73. Turbidity	P, G	Cool, 4°C	48 hours.
Table IC—Organic Tests ⁸			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 66, 88, 89, 92–95, 97. Purgeable Halocarbons.	G, Teflon-lined septum.	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ ..	14 days.
6, 57, 90. Purgeable aromatic hydrocarbons	do	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH ²⁹ .	Do.
3, 4, Acrolein and acrylonitrile	do	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ ; Adjust pH to 4–5 ¹⁰ .	Do.
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96. Phenols ¹¹ .	G, Teflon-lined cap.	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ ..	7 days until extraction, 40 days after extraction.
7, 38. Benzidines ¹¹	do	do	7 days until extraction. ¹³
14, 17, 48, 50–52. Phthalate esters ¹¹	do	Cool, 4°C	7 days until extraction; 40 days after extraction.
72–74. Nitrosamines ^{11,14}	do	Cool, 4°C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ .	Do.
76–82. PCBs ¹¹ acrylonitrile	do	Cool, 4°C	Do.
54, 55, 65, 69. Nitroaromatics and isophorone ¹¹	do	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ store in dark.	Do.
1, 2, 5, 8–12, 32, 33, 58, 59, 64, 68, 84, 86. Polynuclear aromatic hydrocarbons. ¹¹	do	do	Do.
15, 16, 21, 31, 75. Haloethers ¹¹	do	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ ..	Do.
29, 35–37, 60–63, 91. Chlorinated hydrocarbons ¹¹ .	do	Cool, 4°C	Do.
87. TCDD ¹¹	do	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ ..	Do.
Table ID—Pesticides Tests:			
1–70. Pesticides ¹¹	do	Cool, 4°C, pH 5–9 ¹⁵ ..	Do.
Table IE—Radiological Tests:			
1–5. Alpha, beta and radium	P, G	HNO ₃ to pH<2	6 months.

Table II Notes

¹ Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic).

² Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until composting and sample splitting is completed.

³ When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

⁴ Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details. The term "analyze immediately" usually means within 15 minutes or less of sample collection.

⁵ Should only be used in the presence of residual chlorine.

⁶ Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

⁷ Samples should be filtered immediately on-site before adding preservative for dissolved metals.

⁸ Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

§ 136.4

40 CFR Ch. I (7-1-97 Edition)

⁹ Sample receiving no pH adjustment must be analyzed within seven days of sampling.

¹⁰ The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

¹¹ When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

¹² If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

¹³ Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

¹⁴ For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

¹⁵ The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na₂S₂O₃.

¹⁶ Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the 4°C temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature can not be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

[38 FR 28758, Oct. 16, 1973, as amended at 41 FR 52781, Dec. 1, 1976; 49 FR 43251, 43258, 43259, Oct. 26, 1984; 50 FR 691, 692, 695, Jan. 4, 1985; 51 FR 23693, June 30, 1986; 52 FR 33543, Sept. 3, 1987; 55 FR 24534, June 15, 1990; 55 FR 33440, Aug. 15, 1990; 56 FR 50759, Oct. 8, 1991; 57 FR 41833, Sept. 11, 1992; 58 FR 4505, Jan. 31, 1994; 60 FR 17160, Apr. 4, 1995; 60 FR 39588, 39590, Aug. 2, 1995; 60 FR 44672, Aug. 28, 1995; 60 FR 53542, 53543, Oct. 16, 1995]

§ 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure.

(b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

(c) Unless and until printed application forms are made available, an application for an alternate test procedure may be made by letter in triplicate. Any application for an alternate test procedure under this paragraph (c) shall:

(1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Table I.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Analytical Methods Staff, Office of Science and Technology (4303), Office of Water, U.S. Environmental Protection Agency, 401 M Street, SW., Washington, DC 20460. Any application for an alternate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.